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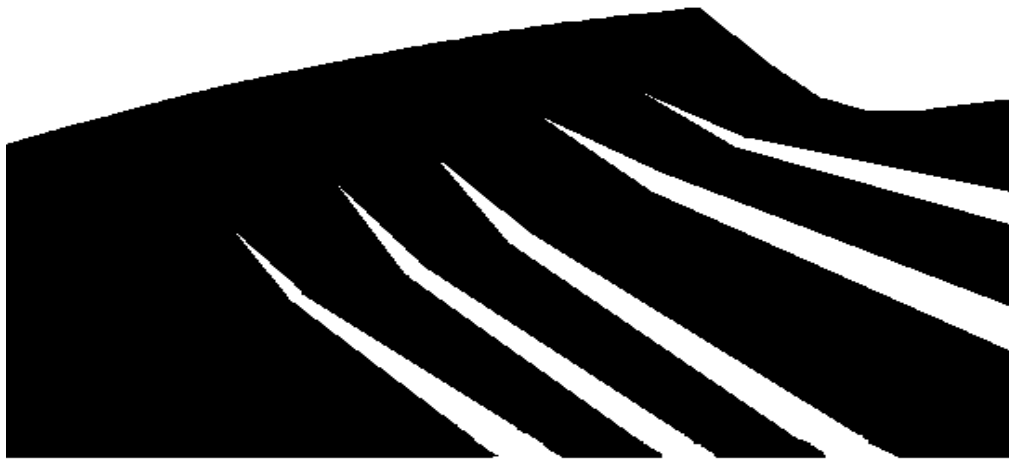
December 10, 1996

LANL-CST-DP-67, R2

Page 1 of 8

ROCK BEAKER EXPERIMENT

LOS ALAMOS QUALITY PROGRAM



APPROVAL FOR RELEASE

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Los Alamos

Yucca Mountain Site

Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0		N/A	Initial procedure.
R1	07/08/94	All	Revised and reformatted to QP-06.3. Revision of this procedure was previously identified as LANL-INC-DP-67.
R2	12/10/96	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

Los AlamosYucca Mountain Site
Characterization Project

ROCK BEAKER EXPERIMENT

1.0 PURPOSE

The purpose of this procedure is to study the diffusion and adsorption of radioisotopes from tracer solutions that contact only rock surfaces under static conditions.

2.0 SCOPE

The procedure describes the steps to assemble a prepared YMP rock beaker within a plexiglas enclosure which minimizes the evaporation of makeup water and the tracer solution.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-INC-DP-60, Preparation of NTS Samples for LANL YMP Solid Core Experiments
LANL-INC-DP-79, Liquid Scintillation Counting of Samples
CST Division Environmental Safety and Health Operational Policy Statement

4.0 DEFINITIONS

4.1 Tracer Solution

The tracer solution is the solution containing the radionuclide(s) that will be studied.

4.2 Sampling Time

Sampling interval for tracer solution and makeup water once the experiment has begun.

4.3 $C_0(t)$

Tracer concentration in initial tracer solution at time t (i.e., the aliquot of the initial tracer solution corresponds to $C_0(t=0)$).

4.4 $C_i(t)$

Tracer concentration in solution I at time t (solution I represents the solution at a given time t_i).

4.5 Representative Sample

A portion of material whose composition and properties are representative of the original sample.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principal Investigator (PI)
- Users of the Detailed Procedure (DP)

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this QP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document or the current work practice is documented in accordance with QP-03.5, Section 6.1.6..
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

The actinides present special problems for studying their sorption and kinetic properties. The actinides exhibit nonselective sorption on nongeologic materials, such as the walls of the sample container. To overcome this inherent difficulty, the sorption of the actinides will be studied as a function of time by using rock beakers so that the actinide will contact only the tuff.

6.2 Equipment and Hardware/Software

- vacuum oven
- balance
- scintillation counter

6.2.1 Equipment Malfunctions

During the process of this experiment, there are no critical equipment malfunctions that are likely to occur that would interrupt the experiment.

6.2.2 Safety Considerations

Ensure compliance with CST Division Environmental Safety and Health Operational Policy Statement.

6.2.3 Special Handling

Ensure that rock beaker is placed in secondary containment such as a laboratory tray. Ensure that the apparatus is placed under a ventilation hood after tracer has been added to the rock beaker.

6.3 Preparatory Verification

Before initiation of the experiment:

- a) Obtain a representative piece of the sample by coring out a cut slab (manipulated according to DP-60). The size of the core (representative piece) can be of any convenient length and diameter (in mm) in the laboratory notebook.
- b) The rock beaker assembly will consist of the sample (manipulated according to DP-60), enclosed in a Plexiglas container (see Attachment 1). Record the unique identifier of the sample used to prepare the rock beaker. If more than one beaker has been prepared from the same sample, an additional alphanumeric character will be added to the identifier in order to maintain uniqueness.
- c) Record the dimensions of the rock beaker to within 0.05 mm. These include length and diameter of the beaker, and depth and diameter of the beaker cavity.
- d) Dry the rock beaker at 30 - 40 °C in a vacuum oven until a constant weight is obtained within 0.01g. Record the dry weight. Record the unique identifier of the balance used.
- e) Equilibrate the beaker with the chosen groundwater until a constant weight is obtained within 0.01 g. Record the groundwater used. Record the wet weight. Record the unique identifier of the balance used.
- f) Assemble the system by placing the rock beaker in the base of the Plexiglas enclosure. Place an O'ring on top of the rock beaker. Use silicone vacuum grease, or equivalent, to seal the Plexiglas container to the cap and to make a seal for the plug. Attach the Plexiglas cap to the container using appropriate screws. Fill both cavities with appropriate makeup water (chosen groundwater).

- g) Prepare the tracer solution. Record the tracer solution used and its reference in the stock solution binder.
- h) Record the sampling time(s) to be used for the experiment.

6.3.1 Hold Points

No hold points are included in this procedure.

6.3.2 Calibration

Balances used for weighing are controlled pursuant to LANL-YMP-QP-12.3. When data are collected from a balance, the unique identifier number of that balance must be recorded in the user's laboratory notebook along with the data collected. Scintillation counter calibrated pursuant to DP-79.

6.3.3 Environmental Conditions

No special conditions are required for this DP. If any special conditions are utilized, record them in the laboratory notebook.

6.4 Control of Samples

- 6.4.1 Samples will be controlled according to QP-08.1.

6.5 Implementing Procedure

6.5.1 Determining Porosity of Representative Sample

6.5.1.1 The representative piece will be used to determine the porosity of the rock beaker sample. Dry representative piece in a vacuum oven at 102 °F. Record dry weight.

6.5.1.2 Equilibrate the representative piece with the chosen groundwater until a constant weight is obtained within 0.01 g. Record the groundwater used. Record the wet weight. Record the unique identifier of the balance used.

- 6.5.2 Remove the plug from the Plexiglas beaker enclosure, empty the cavity of the beaker using a transfer pipet, and dry the cavity of the beaker using a kimwipe.

- 6.5.3 Place an aliquot of the tracer solution in the beaker cavity and replace the plug; also place two aliquots of the tracer solution in scintillation vials and count on scintillation counter to determine the activity of tracer solution at time 0.

NOTE: Evaporation of the tracer solution being equilibrated with the rock beaker can cause errors in the results. Consequently, keep the plug of the beaker securely in place.

6.5.4 Note start time (Julian time).

6.5.5 At intervals specified by the PI, sample the tracer solution in the beaker by removing the plug and transferring two aliquots of the solution to scintillation vials. Note the time at which the sample was taken.

6.5.6 At intervals specified by the PI, sample the solution in the Plexiglas container by removing the plug and transferring two aliquots of the solution to scintillation vials. Note the time at which the sample was taken.

6.5.7 Analyze the tracer in the sampling according to DP-79.

Notebook Entries

The following entries must be made in the laboratory notebook:

- a) Amount of tracer solution placed in the beaker cavity, specifying the units of weight used for measuring amount.
- b) Start time (Julian time).
- c) Amount of tracer solution transferred to the vial(s), specifying the units of weight used for measuring amount.
- d) Julian time at which the sample(s) was taken.
- e) Analytical technique used and amount of tracer determined in the samples taken.

Upon completion of the experiment, verify that no leakage has occurred by visually inspecting the outside of the Plexiglas container. Verify that the information specified in sections 6.3-6.5 has been recorded in the laboratory notebook.

6.6 Data Acquisition and Reduction

The relative concentration of the tracer, $C_i(t)/C_o(t)$, will be calculated for each individual sample.

NOTE: The active recording of data as specified above will constitute the data acquisition. Computer programs such as word processing editors and spreadsheets can be used for recording and formatting data but are not part of the data acceptance criteria.

6.7 Potential Sources of Error and Uncertainty

When work, as described in this DP, cannot be accomplished or would result in an undesirable situation, the work will be stopped and not resumed until this DP is revised to reflect the correct work practice. However, if the revision of this DP cannot be accomplished in a timely manner, the continuation of the work will be described in accordance with QP-03.5

The responsible PI or his/her designee will determine whether to use the data. If a decision to use the data is made, the justification for this decision must be entered in the investigator's logbook or binder.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

- 8.1 Visually inspect for leakage outside of the Plexiglas container. If leakage is detected, the data has to be rejected.
- 8.2 If solution in beaker has higher count than the solution taken at the beginning of the experiment, reject data.

9.0 TRAINING

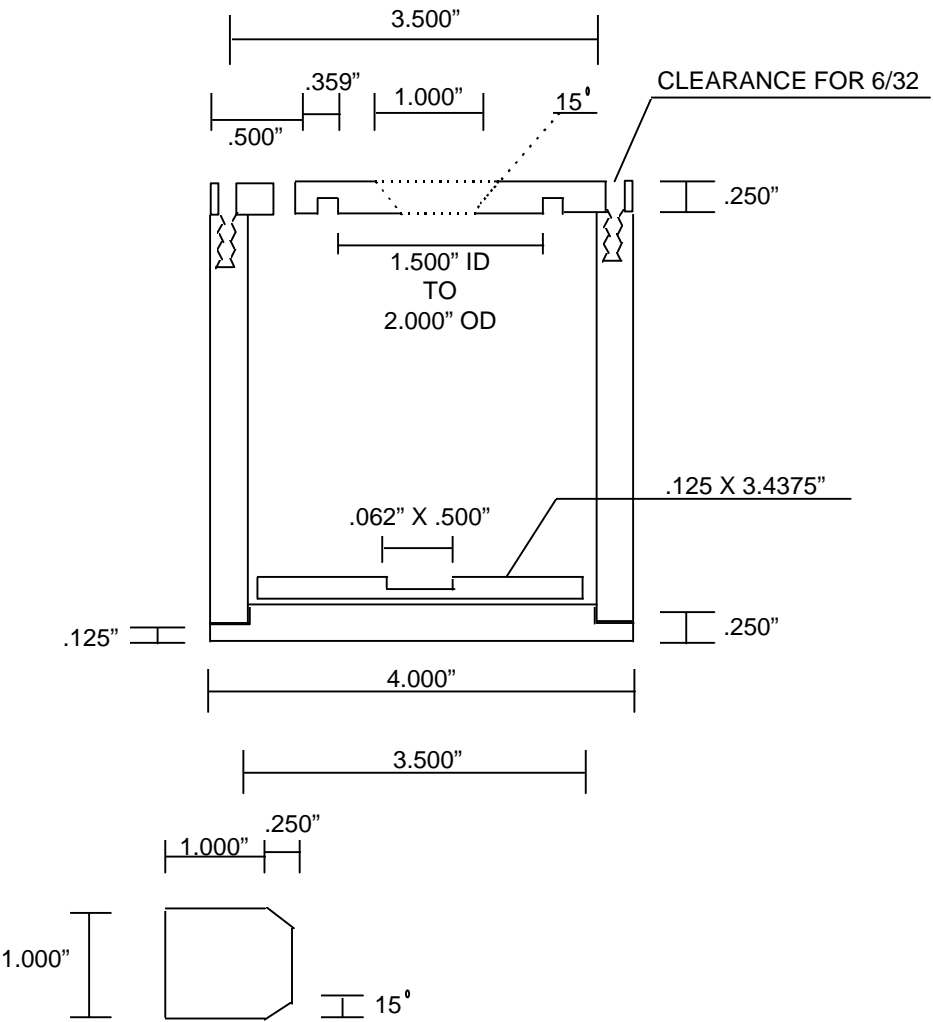
- 9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.
- 9.2 Training to this procedure is accomplished by "read only". Training will be documented in accordance with QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Rock Beaker Assembly

Attachment 2: Example Template for Rock Beaker Experiments

Rock Beaker Apparatus



Example Template for Rock Beaker Experiments

Rock Beaker Experiment Worksheet	
Type of Experiment	
Radionuclide Utilized	
Water Used For Sample Pre Equilibration	
Tracer Sooin Preparation	
Radionuclide	
Oxidation State	
Notebook # for Tracer Solution Preparation	
Notebook Pages for Tracer Solution Preparation	
Concentration of Prepared Tracer Solution	
Unique Tracer Solution Id	
Verification of Liquid Scintillation Counter (a/b) #901133	
ID of Standard Counter	
Total Activity (-Bkg)	
ID of Standard Counted	
Total Activity (-Bkg)	
Procedure Utilized (Uncontrolled)	
Notebook # for Standard Preparation	
Information for Tracer Solution Utilized in	
By Liquid Scintillation Counting	
Unique Tracer Solution Id	
Counter Number	
Region	
Range of Channels	
Date Counted	
Amount Tracer Solution Counted (g)	
Type of Make-Up Water	
Amt of Make-Up Water (ml)	
Count Length	
# of Iterations	
AVG CPM/g of Tracer Solution (-Bkg)	
YMP Balance ID	
Beaker Parameters	
Type of Beaker Material	
Unique Identifier or SMF Id#	
LANL-CST-DP Utilized	
<i>Beaker Dimensions</i>	
Diameter in mm	
Length in mm	
Cavity Diameter in mm	
Cavity Depth in mm	
Representative Place Parameters	
Diameter in mm	

Example Template for Rock Beaker Experiments

Length in mm	
Dry Weight (g)	
Wet Weight (g)	
YMP Balance Utilized	
Unique Id of Water Utilized for Preparation	
Addition of Tracer Solution	
Volume of Tracer Loaded (ml)	
Start Time of Experiment	
YMP Balance Used	
End of Experiment (time)	
pH Measurements	
pH Meter Utilized	
pH Buffers Utilized	
pH Buffer Reading Prior to Making Measurements	
pH Buffer Reading After Making Measurements	
pH of Tracer Solution	
Rock Beaker Comments	
Location of Data	
Raw Experimental Data (notebook #)	
Raw Experimental Data (page (\$) #)	
Reduced Data (optical disk #)	
Reduced Data (subdirectory)	
Reduced Data (file name)	

Example Template for Rock Beaker of Experiments

Sample #	Total weight (g)	Tare weight (g)	Sample weight (g)	Collection time	Total Np CPM in sample (a/b) -Bkg	Np CPM/g- (a/b)- Bkg	Total H3 CPM in sample (a/b)-Bkg	H3 CPM/g (a/b)-Bkg	Cumm. Volume (ml)	C/Co
P1 Bkg					1.8		26.9			
RKBK STD.-1										
RKBK STD.-2										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										

EXAMPLE